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IS 526 (2007): Oil of Palmarosa [PCD 18: Natural and Synthetic Fragrance Materials]

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पामारोजा का तेल — विशिष्टि

(तीसरा पुनरीक्षण)

Indian Standard

OIL OF PALMAROSA — SPECIFICATION

(Third Revision)

ICS 71.100.60

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

June 2007

Price Group 3

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Natural and Synthetic Fragrance Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1954 and subsequently revised in 1968 and 1988. The original standard covered the requirements for the oils of palmarosa and gingergrass but the first revision covered requirements pertaining to oil of palmarosa only. However, analytical characteristics of oil of gingergrass were included in Annex A for information only. Second revision of the standard was carried out in the light of the development of suitable methods of test for detecting possible adulterants in genuine oil of palmarosa. Gas chromatographic analysis has been included for the identity of the oil from the genuine plant materials (*see* Annex A). The requirements for oil of gingergrass as given in the original specification have, however, been included as useful information. This information will serve as a useful basis for comparison to prospective producers interested in genuine oil of palmarosa.

In this revision the requirements (Relative density, Refractive index, Optical rotation and miscibility in alcohol) are reported at 20°C as in International Standards (ISO), besides at 27°C as is in practice at present in the Indian Standards. This would provide basis and facilitate international trade as these would go a long way in removing technical barriers to trade (TBT). This standard contain the flash point of oil of palmarosa as information only. The International Standard on the subject is ISO 4727 : 1988 'Oil of palmarosa [*Cymbopogon martinii* (Roxburgh) W. Watson var. *motia*]'.

Oil of palmarosa, an important perfumery oil, is derived from the plant *Cymbopogon martinii*, stapf, fam. Gramineae and exists in two distinct varieties, namely, *motia* and *sofia*. Oil of palmarosa (ex var. *motia*), is much sought after in perfumery, whereas the comparatively cheaper oil of gingergrass (ex var. *sofia*) is not. It is, therefore, likely that the latter is used to adulterate the former. Also, as the two grasses generally grow side by side, the possibility of inadvertent mixing of the two plant materials cannot be ruled out. Thus, ignorance of the collectors to properly identify, the genuine plant material as well as indiscriminate collection may lead to admixture of *motia* with *sofia* variety. In the context, it may be mentioned that the two varieties of grasses cannot be differentiated morphologically in the dry state. However, they may be distinguished in the field because their forms are different. For example, the leaves of the *sofia* variety form a different angle with the sheath compared to those of *motia*, also the former have a large number of radical leaves than the latter variety (*see* Annex B).

For the purpose of deciding whether a particular requirement of this standard is complied with the values, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard
OIL OF PALMAROSA — SPECIFICATION
(*Third Revision*)

1 SCOPE

This standard prescribes the requirement and methods of sampling and test for oil of palmarosa.

2 REFERENCES

The following standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to invertigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
326	Methods of sampling and test for natural and synthetic perfumery materials:
(Part 1) : 1984	Sampling (*second revision*)
(Part 2) : 1980	Preliminary examination of perfumery materials and samples (*second revision*)
(Part 3) : 2006/ ISO 279 : 1998	Determination of relative density (*third revision*)
(Part 4) : 2005/ ISO 592 : 1998	Determination of optical rotation (*third revision*)
(Part 5) : 2006/ ISO 280 : 1998	Determination of refractive index (*third revision*).
(Part 6) : 2005/ ISO 875 : 1999	Determination of solubility in ethanol (*third revision*)
(Part 7) : 2006/ ISO 1242 : 1999	Determination of acid value (*third revision*)
(Part 8) : 2005/ ISO 709 : 2001	Determination of ester value, content of esters and combined alcohols (*third revision*)
(Part 9) : 1980	Determination of ester value, after acetylation and free alcohols (*second revision*)
(Part 19) : 1998	Gas chromatographic analysis of perfumery materials
1070 : 1992	Reagent grade water (*third revision*)
2284 : 1988	Method for olfactory assessment of natural and synthetic perfumery materials (*first revision*)
6597 : 2001	Glossary of terms relating to fragrance and flavour industry (*second revision*)

3 TERMINOLOGY

For the purpose of this standard, definitions given in IS 6597, shall apply.

4 REQUIREMENTS

4.1 Description

4.1.1 Oil of palmarosa shall be obtained either by water or steam distillation of partially dried leaves, flowers and upper third of the stems, cut at early seed setting stage.

4.1.2 The oil shall be a clear liquid free from sediment, suspended matter, separated water and added adulterants.

4.1.3 The oil shall be examined for its colour, clarity, separated water and sediment as prescribed in IS 326 (Part 2).

4.1.4 The oil shall also be tested olfactory and especially for the by-notes, and for the presence and adulterants and impurities, if any, as prescribed in IS 2284.

4.2 Solubility

The material shall be soluble in 2 volumes of ethanol (70 percent by volume), when tested as prescribed in IS 326 (Part 6).

4.3 Flash Point

The flash point of oil of palmarosa is 93°C. (This value is provided for information only.)

4.4 Chromatographic Profile

Analysis of the essential oil shall be carried out by gas chromatography as per Annex A.

4.5 The material shall be tested for adulteration with oil of gingergrass as prescribed in Annex C.

4.6 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Oil of Palmarosa

(*Clause* 4.6)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to IS (4)
i)	Colour	Light yellow to yellow mobile liquid	326 (Part 2)
ii)	Odour	Rosaceous, with characteristic grassy background	2284
iii)	Relative density[1] :		
	a) at 20°C	0.880 to 0.894	326 (Part 3)
	b) at 27°C	0.876 to 0.888	
iv)	Optical rotation	– 1.4° to + 3°	326 (Part 4)
v)	Refractive index[2] :		
	a) at 20 °C	1.471 0 to 1.478 0	326 (Part 5)
	b) at 27°C	1.470 2 to 1.474 7	
vi)	Acid value, *Max*	1	326 (Part 7)
vii)	Ester value (using about 5 g of the material)	7 to 36	326 (Part 8)
viii)	Ester value after acetylation (using about 1 g of the material)	260 to 280	326 (Part 9)
ix)	Total alcohols[3], calculated, as geraniol (molecular weight 154.2) percent by mass, *Min*	88.8	326 (Part 8)

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[1] The correction factor for relative density for each degree Celsius change in temperature is 0.000 64.

[2] The correction factor for refractive index for each degree Celsius change in temperature is 0.000 38.

[3] The term total alcohol denotes the amount of geraniol present in the free state together with the geraniol, present in the combined state as geraniol esters.

5 PACKING AND MARKING

5.1 Packing

The material shall be supplied in glass bottles, or in suitable containers as agreed to between the purchaser and the supplier. Aluminum containers shall be avoided. The containers shall be tightly closed and nearly full.

5.2 Marking

Each container so filled shall bear legibly and indelibly the following information:

a) Name of the material;

b) Manufacturer's name and address and/or his recognized trade-mark, if any;

c) Net mass of the material;

d) Month and year of manufacture;

e) Batch number; and

f) Cautionary notice, if any.

5.2.1 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material, each not less than 50 ml, shall be drawn as prescribed under **3** of IS 326 (Part 1).

6.2 All the characteristics given in this specification shall be tested on the composite sample. The lot shall be declared as conforming to the requirements of this specification, if all the test results on the composite sample meet the relevant specification requirements.

7 TEST METHODS

7.1 Tests shall be conducted as prescribed under **4** and the appropriate references to relevant parts of these standards are given in col 4 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Foreword* and *Clause* 4.4)

GAS CHROMATOGRAPHIC ANALYSIS FOR IDENTIFICATION OF OIL OF PALMAROSA

A-1 GENERAL

The chromatographic conditions given here are for information and guidance.

A-1.1 Outline of the Method

A sample of the material is dissolved in a suitable solvent (for example, hexane, cyclohexane or acetone) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

A-2 PROCEDURE

A-2.1 The analysis shall be done as per IS 326 (Part 19). The typical chromatogram for oil of palmarosa in capillary column with the following chromatographic conditions is shown in Fig. 1 and Fig. 2.

A-2.1.1 *Gas Chromatographic Conditions on Polar Column*

Column	PE-Wax (Polyethylene glycol)
a) Size	Length – 30 m
	Internal Diameter 0.32 mm
b) Film thickness	0.25 µm
Stationery phase	Polyethylene glycol, 20M
Conditions	
Oven temperature	50°C to 220°C
Initial time	4 min
Final hold time	3.5 min
Split flow rate	30ml/min
Injector port temperature	220°C
Carrier gas	Hydrogen@7psi
Volume injected	0.2 µl
Detector	
a) Type	Flame ionization detector
b) Temperature	250°C

A-2.1.2 *Gas Chromatographic Conditions on Non-polar Column*

Column	95 percent dimethyl polysiloxane
a) Size	Length – 60 m
	Internal diameter 0.32 mm
b) Film thickness	0.25 µm
Conditions	
Oven temperature	70°C to 250°C
Initial time	2 min
Final hold time	2 min
Split flow rate	30ml/min
Injector port temperature	220°C
Carrier gas	Hydrogen@10psi
Volume injected	0.2 µl
Detector	
a) Type	Flame ionization detector
b) Temperature	280°C

A-2 CALCULATION

A-2.1 Area Measurements (*see* Note)

Since normal peaks approximate a triangle, the area is measured by multiplying the peak height times the width of the half height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

NOTE — Other methods of area measurements, namely, triangulation, disc integrator and electronic digital integrator, if fixed with GC machine, would be of great advantage.

A-2.2 Area Normalization (*see* Note)

By normalizing, it is meant calculating the area of each and dividing the individual areas by total areas,

$$\text{Percentage of } A = \frac{\text{Area of } A \times 100}{\text{Total area}}$$

NOTE — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

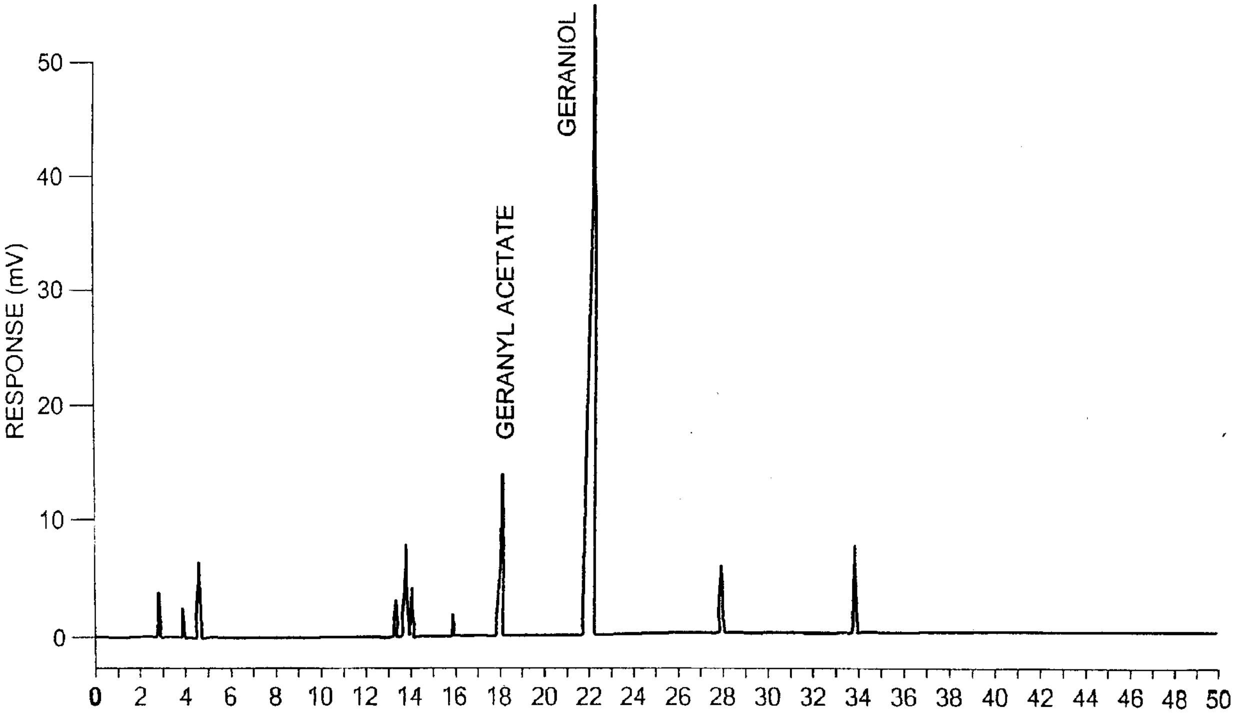

FIG. 1 A TYPICAL CHROMATOGRAM FOR OIL OF PALMAROSA ON POLAR COLUMN

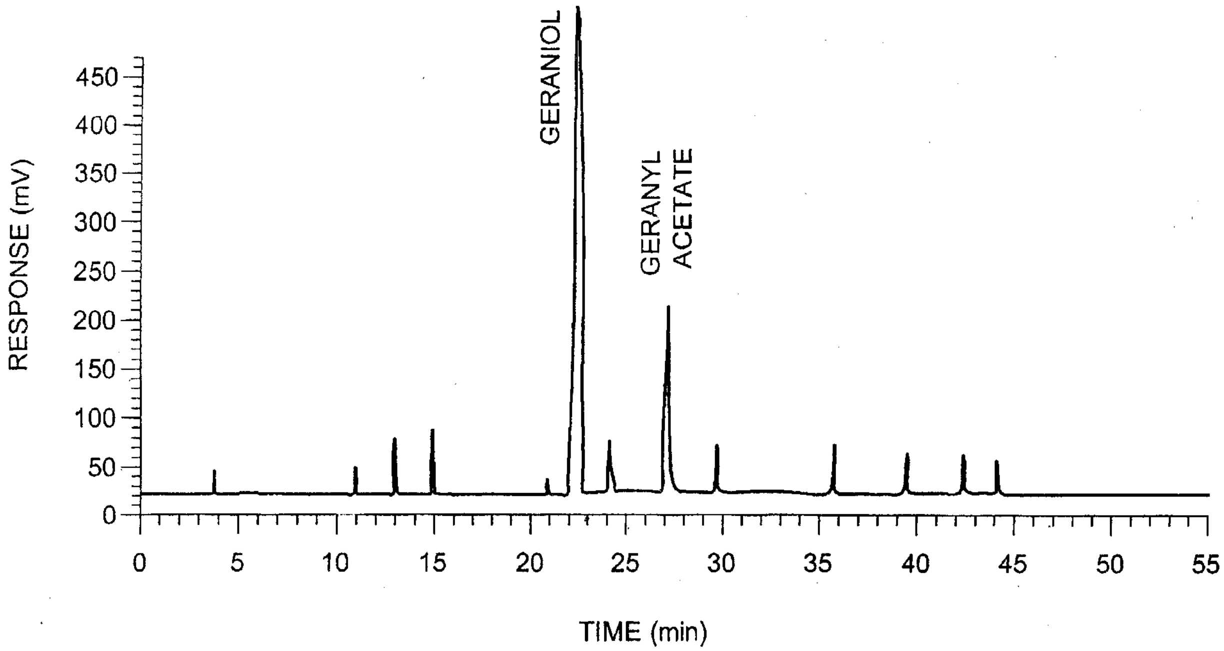

FIG. 2 A TYPICAL CHROMATOGRAM FOR OIL OF PALMAROSA ON A NON-POLAR COLUMN

ANNEX B
(*Foreword*)

ANALYTICAL CHARACTERISTIC OF OIL OF GINGERGRASS DISTILLED FROM LEAVES, FLOWERS AND UPPER PART OF STEMS OF *CYMBOPOGON MARTINI* STAPF. VAR. *SOFIA* FAM. GRAMINEAE

Sl No. (1)	*Characteristic* (2)	*Requirement* (3)	*Method of Test, Ref to IS* (4)
i)	Colour and appearance	Brownish yellow to brownish red, hazy	326 (Part 2)
ii)	Odour	Sharp but pleasant odour	2284
iii)	Relative density[1], at 20°C	0.902 5 to 0.931 5	326 (Part 3)
iv)	Optical rotation	– 14° to + 54°	326 (Part 4)
v)	Refractive index[2], at 20°C	1.477 6 to 1.492 6	326 (Part 5)
vi)	Acid value, *Max*	6	326 (Part 7)
vii)	Ester value	13.5 to 34	326 (Part 8)
viii)	Ester value after acetylation	140 to 180	326 (Part 9)
ix)	Total alcohols, calculated, as geraniol percent by mass	36 to 60	326 (Part 8)

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[1] The correction factor for relative density for each degree Celsius change in temperature is 0.000 64.

[2] The correction factor for refractive index for each degree Celsius change in temperature is 0.000 38.

ANNEX C
(*Clause* 4.5)

DETECTION OF ADULTERATION OF OIL OF PALMAROSA WITH OIL OF GINGERGRASS

C-1 OUTLINE OF THE METHOD

Oils of palmarosa and gingergrass produce different colour reactions with different reagents. This is used for qualitative detection of the admixture of the two oils.

C-2 REAGENTS

C-2.1 Ethanolic Solution of Phloroglucinol — To 1 ml of 10 percent (*m/v*) ethanolic solution of phloroglucinol, add 9 ml of concentrated hydrochloric acid .

C-2.2 Ethanolic Solution of Vanillin — To 1 ml of 20 percent (*m/v*) ethanolic solution of vanillin, add 9 ml of concentrated hydrochloric acid.

C-2.3 Ethanol Solution of Phosphomolybdic Acid — To 10 ml of 10 percent (*m/v*) ethanolic solution of phosphomolybdic acid, add 5 ml of concentrated hydrochloric acid.

NOTE — Ethanol used in the preparation of ethanolic solutions of the three reagents shall be aldehyde free.

C-3 PROCEDURE

Place a drop of the oil on a strip of filter paper followed by a drop of the reagent directly over them. Observe the colour developed in the first few seconds and subsequent change in colour during the next 5 min. Carry out the test with all the three reagents on the oil.

C-4 REACTIONS

The oils of palmarosa and gingergrass, when pure, develop colours as indicated in Table 2.

Table 2 Colours Developed with the Pure Oils of Palmarosa and Gingergrass

(*Clause* C-4)

Sl No.	Reagent	Colour Observed with	
		Oil of Palmarosa	Oil of Gingergrass
(1)	(2)	(3)	(4)
i)	Ethanolic solution of phloroglucinol	Reddish-brown, changes to pink in 2 min	Yellow, changes immediately to light brownish yellow, becomes lighter in intensity after 5 min
ii)	Ethanolic solution of vanillin	Light-purple, changes to greyish yellow after 5 min	Pink within few seconds, remains unchanged for 5 min
iii)	Ethanolic solution of phosphomolybdic acid	Yellowish-green after 30 second, changes to dirty green in 3 min acquires a blue tinge after 5 min	Light green, changing greenish-blue, becomes bluish-black after 2 min

GMGIPN—108 BIS/ND/07—300

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This Indian Standard has been developed from Doc : No. PCD 18 (2306).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Typeset by **PCAT**, Delhi

PRINTED BY THE GENERAL MANAGER GOVERNMENT OF INDIA PRESS, NASHIK